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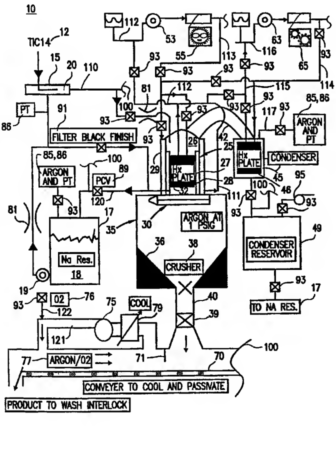

(57) Abstract: A system, method and apparatus for treating a slurry of liquid metal and solid particles is disclosed using various combinations of vacuum distillation and transfer with hot inert gas. Various mechanisms are disclosed for sealing inerted or vacuum chambers to prevent oxygen infiltration

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SEPARATION SYSTEM, METHOD AND APPARATUS

RELATED APPLICATIONS

This application, pursuant to 35 U.S.C. Section No.119(e) and any other applicable provision of Title 35 U.S.C., claims the priority benefit of U.S. Provisional Application Serial No. 60/499,857, Filed September 3, 2002 entitled: SEPARATION SYSTEM, METHOD AND APPARATUS and PCT Application Serial No. PCT/US03/027649 filed September 2, 2003 entitled SEPARATION SYSTEM AND PROCESS by Richard Armstrong and Lance Jacobsen and of U.S. Provisional Application Serial No. 60/497,192 filed August 22, 2003 entitled INDEXING SEPARATION SYSTEM by William A. Ernst and PCT patent application Serial No PCT/US03/027653 filed September 3, 2003 entitled: FILTER CAKE TREATMENT APPARATUS AND METHOD by Richard Anderson, Donn Armstrong and Lance Jacobsen and PCT Application Serial PCT/US03/027647 filed September 3, 2003 entitled: FILTER EXTRACTION MECHANISM by Richard Anderson, Donn Armstrong and Lance Jacobsen.

BACKGROUND OF THE INVENTION

This invention relates to a separation system, method and apparatus useful for separating the slurry produced by Armstrong method as disclosed and claimed in U.S. Patents 5,779,761; 5,958,106 and 6,409,797, the disclosures of each and every one of the above-captioned patents are incorporated by reference.

Although various metals of controlling the reaction temperature are disclosed in the Armstrong Process, the most commercially advanced method, at the present time, is the use of excess reductant metal, such as sodium, to absorb the heat of reaction during the exothermic reduction of the halide gas, such as titanium tetrachloride to product titanium or a

combination of chlorides to produce an alloy. Using an excess of liquid reductant metal, there is produced as reaction products, a powder of the element or alloy to be produced, a particulate salt and excess reductant metal. It should be understood that the scope of this invention is beyond the product of the Armstrong Process and extends to any slurry composed of liquid metal and particulates in which the particulates have to be separated from the liquid metal and thereafter treated. For purposes of brevity only, but not by way of limitation, the description will be in terms of the exothermic reduction of titanium tetrachloride with sodium to produce titanium particles, sodium chloride particles and excess sodium.

During the commercialization of the Armstrong Process, in particular, there is a requirement to handle expeditiously the product from the Armstrong reactor, because the product is formed so quickly the continuous processing thereof has become extremely important. The present invention provides a system and method for handling the product produced by the Armstrong Process such that the Armstrong reactor may be operated continuously to produce 2,000,000,000 pounds per year of elemental metal or alloy while utilizing a single separation vessel and system for the reactor. This is important because it permits the Armstrong reactor to be operated twenty-four hours a day, 7 days a week, economically to produce metal or alloys or any material made by the Armstrong Process as well as applying to handling other slurries as hereinbefore stated.

In the production of a metal or alloy or other elemental material as described in the above-referenced patents, a slurry is produced which if filtered provides a filter cake in the form of a gel. The slurry has a solids fraction which depends in large part on the amount of excess reductant metal used to control the steady-state temperatures at which the reaction runs. As liquid metal drains through the filter to build the filter cake, a gel is formed from which particles do not settle, unless the gel is broken, such as by mechanical disturbance or

other means. The gel when formed includes the metal particles formed during the reduction, the salt particles formed during the reduction and interstitial liquid metal. The liquid metal in the gel has to be removed by way of distillation with or without a vacuum or by contact with a hot sweep gas, preferably inert to the constituents of the gel with or without a vacuum or any combination thereof.

Summary of the Invention

A principal object of the invention is to provide a separation system, method and apparatus for the Armstrong process disclosed in the '761, '106 and '797 patents;

Another object of the invention is to provide a continuous separation system.

The invention consists of certain novel features and a combination of parts hereinafter fully described, illustrated in the accompanying drawings, it being understood that various changes in the details may.be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

Brief Description of the Drawings

Figure 1 is a schematic illustration of the separation system of the present invention.

FIG. 2 is a schematic view of a system for practicing the method of the present invention;

FIG. 3 is an enlarged schematic representation of the product filtering disc portion of the system illustrated in Fig. 2shown in longitudinal sectional view;

FIG. 4 is a horizontal cross-sectional view of the vessel illustrated in Fig. 3

FIG. 5 is an alternate embodiment of the system illustrated in Fig. 2 and

FIG. 6 is a schematic representation of a variety of processes and products made by or from powder separated from slurries according to the present invention.

FIG. 7 is a graph of pressure rise versus time for a flat plate filter nutsche runs;

FIG. 8 shows data for various temperatures as a function of time and pressure;

FIG. 9 shows a schematic of the filter trap for the above example; and

FIG. 10 shows a schematic of another embodiment of the filter trap of Fig. 3.

FIG. 11 is a schematic diagram showing the two vessels and an embodiment of the transfer mechanism therebetween;

FIG. 12 is a schematic of an alternate embodiment of the present invention; and

FIG. 13 is a schematic illustration of yet another embodiment of the present invention; and

FIG. 14 is a schematic representation of a separation system incorporating features of Figs. 1-13.

Detailed Description of the Invention

The system 10 of the present invention deals with the separation of a metal, alloy or ceramic product, such as titanium, for example only, from the reaction products in the Armstrong process. Although the Armstrong process is applicable to a wide variety of exothermic reactions, it is principally applicable to metals, mixtures, alloys and ceramics disclosed in the above-mentioned patents. The product of Armstrong process is a slurry of excess reductant metal, product metal and alloy or ceramic and salt produced from the reaction. This slurry has to be separated so that various parts of it can be recycled and the produced metal, alloy or ceramic separated and passivated if necessary.

Turning now to the schematic illustration of the system and process of the present invention illustrated in Fig. 1, there is disclosed in the system 10 a source of, for illustration purposes only, titanium tetrachloride 12 which is introduced into a reactor 15 of the type hereinbefore disclosed in the Armstrong process. A supply tank or reservoir 17 with a supply of sodium (or other reductant) 18 is transferred by a pump 19 to the reactor 15 wherein a

slurry product 20 of excess reductant and metal, alloy or ceramic, and salt is produced at an elevated temperature, all as previously described in the incorporated patents.

The slurry product 20 is transferred to a vessel 25 which is in the illustration dome-shaped, but not necessarily of that configuration, the vessel 25 having an interior 26 into which the slurry product 20 is introduced. A filter 27, preferably but not necessarily cylindrical, is positioned within the interior 26 and defines an annulus 28, the slurry product 20 being received inside the cylindrical filter 27. An annular heat exchanger 29 is positioned around the vessel 25, all for a purpose hereinafter disclosed.

The vessel 25 further includes a moveable bottom closure 30. Heat exchange plates 32 are connected as will hereinafter be described to an isolated heating system 50. A collection vessel 35 is positioned below the vessel 25 and is sealed therefrom by the moveable bottom closure 30. The collection vessel 35 has an inwardly sloping bottom surface 36 which leads to a crusher 38 and a valve 39 in the outlet 40 of the collection vessel 35.

Finally, a vapor conduit 42 interconnects the top of the vessel 25 and particularly the interior 26 thereof with a condenser vessel 45, the condenser vessel having a heat exchange plate 46 connected, as hereinafter described, to an isolated cooling system 60. The condenser 45 is connected to a condenser reservoir 49, the condensate collected therein being routed to the sodium supply tank or reservoir 17.

The isolated heating system 50 includes a head tank 52 for the heating fluid which is moved by pump 53 to the heater 55 as will be hereinafter described, connected to both the heat exchanger 29 surrounding the vessel 25 and the heat exchange plates 32 interior of the vessel 25. The isolating cooling system 60 also is provided with a head tank 62, a pump 63

and a cooler 65 which serves to cool the cooling fluid circulated in an isolated loop to the cooling plates 46 as will be hereinafter set forth.

Below the valve 39 and the collection vessel 35 is a product conveyor 70 having a baffle or cake spreader 71 extending downwardly toward the conveyor 70. The conveyor 70 onto which the produced metal, alloy or ceramic and salt are introduced from the collection vessel 35, after removal of the excess reductant metal, is contacted with a counter current flow of gas, preferably but not necessarily oxygen and argon, 77 from a blower 75 in communication with a supply 76 of oxygen and the supply of inert gas such as argon. The heat exchanger 79 is in communication with the blower 75 so as to cool the oxygen/argon mixture 77 as it flows in counter current relationship with the produced metal, alloy or ceramic on the conveyor 70, thereby to contact the product particulates with oxygen to inert the produced metal, alloy or ceramic when required but not so much as to contaminate the produced material.

As indicated in the flow sheet of Fig. 1, there are a plurality of flow meters 81 distributed throughout the system, as required and as well known in the engineering art. There are pressure transducers 86 and pressure control valves 89 where required, all within the engineering skill of the art. A back filter valve 91 is provided in order to flush the filter 27 if necessary. Additionally, a variety of standard shut-off valves 93 are positioned within the loop, hereinafter to be explained and as required. A vacuum pump 95 is used to draw a vacuum in the vessel 25, as will be explained, and the symbol indicated by reference numeral 100 indicates that a plurality of the same or similar systems may be operating at any one time, it being remembered that the enclosed figure is for a single reactor 15 and one separation vessel 25, wherein as in a commercial production plant, a plurality of reactors 15 may be

operating simultaneously each reactor 15 may have more than one separation vessel 25, all depending on engineering economics and ordinary scale up issues.

Product 20 from the reactor 15 exits through line 110 and enters vessel 25 at the top thereof. Although line 110 is shown entering above the filter 27, preferably the line 110 and filter 27 are positioned so that slurry 20 is introduced below the top of filter 27 or in the center of the filter or both. As described in the previously incorporated patents, the slurry product 20 consists of excess reductant metal, salt formed by the reaction and the product of the reaction which in this specific example is titanium existing as solid particles. The product 20 in slurry form from the reactor 15 is at an elevated temperature depending on the amount of excess reductant metal present, the heat capacity thereof and other factors in the reactor 15 during operation of the Armstrong process. In the vessel 25 is a filter 27 which occupies a portion of the interior 26 of the vessel 25, the interior optionally being heated with the annular heat exchanger 29. The slurry product 20 is directed to the interior of the filter 27 where the slurry contacts the heat exchange plates 32.

In the heating system 50, the heat exchange fluid in the plates 32 pass with the heat exchange fluid from the annular heat exchanger 29 through line 111 to the line 112 which connects the heat exchange medium supply in the head tank 52 to the heat exchanger 55. Fluid moves from the heater 55 through the heat exchange plates 32 by means of the pump 53 as the heated heat exchange fluid flows out of the heat exchanger 55 through line 113 and back into the heat exchange plates 32 and/or the annular heat exchanger 29. Because the heating system 50 is a closed loop, the heat exchange fluid may or may not be the same as the reductant metal used in the reactor 15. NaK is shown as an example because of the low melting point thereof, but any other suitable heat exchange fluid may be used. Suitable valves 93 control the flow of heat exchange fluid from the heater 55 to either or both of the

heat exchanger 29 and plates 32. Preferably, the plates 32 are relatively close together, on the order of a few inches, to provide more heat to the cake which forms as excess reductant metal vaporizes. Moreover, closer plates 32 reduce the path length the heat has to travel and the path length the excess reductant metal vapor travels through the forming cake, thereby to reduce the time required to distill and remove excess reductant metal from the vessel 25. Exact spacing of the plates 32 depends on a number of factors, including but not limited to, the total surface area of the plates, the heat transfer coefficient of the plates, the amount of reductant metal to be vaporized and the temperature differential between the inside and the outside of the plates.

When the slurry product 20 comes out of the reactor 15, it is at a pressure at which the reactor 15 is operated, usually up to about two atmospheres. The product slurry 20 enters the inside of filter 27 under elevated pressure and gravity results in the liquid reductant metal being expressed through the filter 27 into the annular space 28 and fed by the line 120 into the reservoir 17. The driving force for this portion of the separation is gravity and the pressure differential between the reactor 15 and the inlet pressure of pump 19. If required the annulus 28 may be operated under vacuum to assist removal of liquid reductant metal, or the pressure in vessel 25 may be increased during the deliquoring of the reductant metal. After sufficient liquid metal has drained through the filter 27 by the aforementioned process, the PCV valve 89 is closed and other valves 93 are closed to isolate vessel 25 and then the valve 93 to the vacuum pump 95 is opened, whereupon a vacuum is established in the interior 26 of vessel 25. Heating fluid (liquid or vapor, for instance Na vapor) is directed into the heat exchanger plates 32 to boil the remaining reductant metal 18 producing a filter cake. The temperature in vessel 25 is elevated sufficiently to vaporize remaining liquid metal reductant 18 therein which is drawn off through conduit 42 to the condenser 45. The conduit 42 is required to be

relatively large in diameter to permit rapid evacuation of the interior 26 of the vessel 25. Because the pressure drop between the vessel 25 and the condenser 45, during vaporization of the reductant metal 18 is low, the specific volume is high and the mass transfer low, requiring a large diameter conduit 42. Boiling the reductant metal on the shell side is accomplished by heat exchange with a heated fluid on the tube side.

The annular heat exchanger 29 is optionally operated to maintain the expressed liquid in the annulus 28 at a sufficient temperature to flow easily and/or to provide additional heat to the vessel 25 to assist in vaporization of excess reductant metal from the interior 26 thereof. After liquid metal reductant vapor has been removed from the interior 26 of the vessel 25, a filter cake remains from the slurry 20. The appropriate valves 93 are closed and the vacuum pump 95 is isolated from the system.

In the condenser 45, heat exchange plates 46 are positioned in order to cool the reductant metal vapor introduced thereinto. The cooling system 60 is operated in a closed loop and maintained at a temperature sufficiently low that reductant metal vapor introduced into the condenser 45 condenses and flows out of the condenser, as will be disclosed. The cooling system 60 includes a cooler 65 as previously described and the pump 62. The coolant exits from the cooler 65 through line 114 which enters the heat exchange plates 46 and leaves through a line 115 which joins the line 116 to interconnect the head tank 62 and the cooler 65. As seen in the schematic of Fig. 1, the heat exchange fluid used in the heating system 50 and the cooling system 60 may be the same or may be different, as the systems 50 and 60 can be maintained separately or intermixed.

Both the vessel 25 and the condenser 45 are operated at least part of the time under a protective atmosphere of argon or other suitable inert gas from the argon supply 85, the pressure of which is monitored by the transducer 86, the (argon) supply inert gas 85 being

connected to the condenser 45 by a line 117, the condenser 45 also being in communication with the vessel 25 by means of the oversized conduit 42. Further, as may be seen, each of the heating system 50 and the cooling system 60 is provided with its own pump, respectively 53 and 63. As suggested in the schematic of Fig. 1, the heating and cooling fluid may, preferably be NaK due to its lower melting point, but not necessarily, and as an alternative could be the same as the reductant metal in either liquid or vapor phase, as disclosed.

After sufficient reductant metal 18 has been removed from the slurry 20, via the filter 27 and the conduit 42, remaining therein is a combination of the titanium product in powder form and salt made during the exothermic reaction in reactor 15. Because the resultant dried cake has a smaller volume than the slurry product 20 introduced, when the movable bottom closure 30 is opened, the dry cake falls from the filter 27 into the collection vessel 35 whereupon the combination of salt and titanium fall into the crusher 38 due to the sloped bottom walls 36. In the event the cake does not readily fall of its own accord, various standard vibration inducing mechanism or a cake breaking mechanism may be used to assist transfer of the cake to the collection vessel 35. The collection vessel 35 as indicated is maintained under an inert atmosphere at about atmospheric pressure, and after the cake passes through the crusher 38 into the exit or outlet 40, the cake passes downwardly through valve 39 onto the conveyor 70. There is a cake spreader or baffle 71 downstream of the valve 39 which spreads the cake so that as it is contacted by a mixture 77 of inert gas, preferably argon, and oxygen flowing counter-current to the direction of the product, the titanium powder is passivated and cooled. Although the conveyor 70 is positioned in Fig. 1 horizontally, it may be advantageous to have the conveyor move upwardly at a slant as a safety measure in the event that closure 30 fails, then excess reductant metal would not flow toward a water wash.

In addition, there may be cost advantages in having the product wash equipment on the same level as the separation equipment.

Cooling and passivating is accomplished in the cooler 79 with blower 75 which blows a cooled argon and oxygen mixture through a conduit 121 to the product, it being seen from the schematic that the counter-current flow of argon and oxygen with the product has the highest concentration of oxygen encountering already passivated and cooled titanium so as to minimize the amount of oxygen used in the passivation process. Oxygen is conducted to the system from a supply thereof 76 through a valve 93 and line 122 and is generally maintained at a concentration of about 0.1 to about 3% by weight. The mixture of passivated titanium and salt is thereafter fed to a wash system not shown. Various flow meters 81 are positioned throughout the system as required, as are pressure control valves 89 and pressure transducers 86. A filter backwash valve 91 is positioned so that the filter 27 can be backwashed when required if it becomes clogged or otherwise requires backwashing. Standard engineering items such as valves 93, vacuum pump 95 and pressure transducers 86 are situated as required. Symbol 100 is used to denote that parallel systems identical or similar to all or a portion of the system 10 illustrated may be operated simultaneously or in sequence.

In the Armstrong process, the production of the metal, alloy or ceramic is continuous as long as the reactants are fed to the reactor. The present invention provides a separation system, apparatus and method which permits the separation to be either continuous or in sequential batches so rapidly switched by appropriate valving as to be as continuous as required. The object of the invention is to provide a separation apparatus, system and method which allows the reactor(s) 15 in a commercial plant to operate continuously or in economic batches. Reduction of the distillation time in vessel 25 is important in order to operate a plant economically, and economics dictate the exact size, number and configuration of separation

systems and production systems employed. Although described with respect to Ti powder, the invention applies to the separation of any metal, alloy thereof or ceramic produced by the Armstrong process or other industrial processes.

The heating mechanism shown is by fluid heat exchange, but heaters could also be electric or other equivalent means, all of which are incorporated herein. The bottom closure 30 is shown as hinged and is available commercially. The closure 30 may be clamped when shut and hydraulically moved to the open position; however, sliding closures such as gate valves are available and incorporated herein. Although the reactor 20 is shown separate from the vessel 25, the invention includes engineering changes within the skill of the art, such as but not limited to incorporating reactor 20 into vessel 25. Although not shown in Fig. 1, it is contemplated that the slurry as it forms a cake on the filter may be agitated and the cake may be broken to facilitate distillation and/or transfer. Although vessel 35 is illustrated in one embodiment, the vessel 35 could easily be designed as a pipe or the like. Also, the crusher 38 could be located in vessel 25 or intermediate vessel 25 and vessel 35. Moreover, the cake forming on the filter 27 may be broken up prior to or during or subsequent to removal of the liquid metal therefrom. Similarly, when referring to an inert environment, the invention includes a vacuum as well as an inert gas. An important feature of the invention is the separation of vessels 25 and 35 so the environments of each remain separate. That way, no oxygen can contaminate either vessel.

In one specific example, a reactor 15 producing 2 million pounds per year of titanium powder or alloy powder requires two vessels 25, each roughly 14' high and 7' in diameter with appropriate valving, so that the reactor 15 would operate continuously and when one vessel 25 was filled, the slurry product from the reactor would switch automatically to the second

vessel 25. The fill time for each vessel 25 is the same or somewhat longer than the deliquor, distill and evacuation time for vessel 25.

Changing production rates of reactor 15 simply requires engineering calculations for the size and number of vessels 25 and the related equipment and separation systems. The invention as disclosed permits continuous production and separation of metal or ceramic powder, while the specific example disclosed permits continuous separation with two or at most three vessels 25 available for each reactor 15. With multiple reactors 15, the number of vessels 25 and related equipment would probably be between 2 and 3 times the number of reactors.

Referring to Figures 2-4, there is disclosed a system 10A for continuously processing a liquid metal slurry containing particulates. In this description, powder and particulates are used interchangeably. More particularly, the system 10A includes a reactor 11 such as, but not limited to the type shown in the Armstrong Process, including a nozzle12 through which liquid metal flows and having a housing 14 surrounding the nozzle. A gas inlet 15 serves to introduce gas from a source 16 thereof into the liquid metal thereby producing an exothermic reaction as described in the referenced Armstrong patents. The product from the exothermic reaction may be a slurry of a liquid reducing metal, such as sodium, having dispersed therein particles of the element or alloy produced, such as titanium or an alloy thereof, and the reaction product from the gas, which may be sodium chloride or combination of chloride salts, as in the case of sodium and titanium tetrachloride. The slurry leaves the reactor housing 14 through an outlet 18 and is introduced into a receiving vessel 20 having near the top thereof a dome portion 21 and a cylindrical portion 22 terminating in a frustoconical portion 23 having a discharge outlet 25 at the bottom thereof terminating in a circular flange 26. A motor 30 may be mounted at the top of the vessel 20 connected to an output shaft 31

having an agitator 32 at the bottom of the cylindrical portion 22 or the frustoconical portion 23 as illustrated, for a purpose hereinafter set forth.

Indexing filter system 35, as seen particularly in Fig. 3, is in communication with the vessel 20 and more particularly includes a housing 36, having a top 37, a cylindrical side wall 38 provided with opposed upper apertures 39 and opposed lower apertures 41. The housing 36 is also provided with an outlet 43 having a conduit 44 extending therefrom, for a purpose hereinafter set forth.

An index drive 45 includes a motor 46 having an output shaft 47 in communication with a clutch mechanism 48 connected to an axle 49, the end of which rests in a bearing 51. An aperture 49A is provided in the cylindrical wall 38 to accommodate the axle 49.

An indexing disc 55 is rotatably mounted on the axle 49, the disc having a plurality of longitudinally spaced apart chambers 56 therein, six such chambers being shown for purposes of illustration.

A filter 60, preferably but not necessary a metal wedge wire, is positioned in exit conduit 65 and has a collar 61 maintained in sealing contact with the disc 55 through a spring and pin arrangement 62. The chambers 56 in the disc 55 are also in contact with a collar and spring and pin arrangement in communication with an inlet conduit 63 so as to provide a sealing arrangement for each chamber 56 as it rotates about the axle 49. There is, as illustrated in Fig. 3, a T-shaped conduit 66 having flanges and seals 67 connecting the outlet 25 of the vessel 20 to the indexing disc 55, there being provided a sealing flange 26A (Fig. 2) with the usual seals (not shown) to provide a suitable connection between the vessel 20 and the indexing filter system 35.

Compaction ram assembly 70 is mounted to the housing 36 of the indexing filter system 35 and includes a piston rod 71 having mounted thereon a piston 72. The piston rod

71 is surrounded by a bellows seal 73 and is connected at one end to a suitable drive or motor assembly 74 for longitudinal movement toward and away from the chamber 56, as will hereinafter be described.

A discharge ram assembly 80 is mounted to the housing 36 including a similar piston rod 82, bellows seal 83 and drive motor 84 which is similar to the compaction ram assembly 70. In the illustration of Fig. 3, the discharge ram assembly 80 is rotated for clarity but may be positioned anywhere around the housing 36 in which a chamber 56 is positioned during the indexing of the disc 55, as is well understood by any competent engineer. Moreover, the system 10A of the present invention may also include two or more compaction ram assemblies and two or more discharge ram assemblies, each version being a matter of choice of design, well within the skill of the art. The discharge ram assembly 80 further includes, as did the compaction ram assembly 70, collars 86 and springs and retaining pins 87 to ensure a seal between the discharge ram assembly 80, the indexing disc 55 and the outlet conduit 90 which is surrounded by an outer containment tube or conduit 91, for a purpose hereinafter described.

A cake breaker 93 which may be in the form of a stationary grid or flexible members is positioned at the end of the distillation system 95, as will be explained and for a purpose hereinafter described.

A distillation system 95 is in communication with the outlet conduit 90 of the indexing filter system 35 and includes a longitudinally extending conveyor 96 in a container 98 having a heat exchanger 97 in heat exchange relationship therewith. The distillation system 95 is in communication with a condenser assembly 100 by means of one more tubes 101 extending from the container 98 to a condenser container103, it being understood that the condenser container 103 is shown for purposes of illustration as an elongated container but

may be of any size or shape as determined. The condenser container 103 is also connected by the conduit 44 to the outlet 43 of the housing 36. A conduit 102 provides communication between the condenser assembly 100 and a liquid metal supply 105 in the form of vessel having connected thereto a distillation vacuum pump 106 and an outlet pipe 107. A pump108 pumps liquid metal from the supply vessel 105 through a conduit 109 to a liquid metal accumulation tank 115. The tank 115 also receives liquid metal from a head tank 110 in communication with the outlet conduit 65 from the indexing filter system 35, the head tank 110 being in communication with the accumulation tank 115 by means of a conduit 113. A heat exchanger 112 may be in heat exchange relationship with the outlet conduit 65 if heat is needed to be added or removed from the liquid metal exiting the indexing filter system 35, as will be explained.

A vessel 120 is positioned in communication with the distillation system 95 and includes a valve 121, the vessel 120 being in communication with a pump 122 which in turn is in communication with a vessel 125 or lock hopper which is also provided with a valve 126 at the bottom thereof. The vessel or lock hopper 125 through valve 126 is in communication with a passivation system 130 which includes a containment vessel 131, a conveyor 132 in communication with a gas inlet conduit 133 and a gas outlet conduit 134 in communication with a pump 135. The passivation system 130 has an outlet 136, all for a purpose hereinafter set forth.

Operation of the system 10A is as follows. A supply of gas 16 is brought to temperature in a boiler which is meant to be included in the vessel 16 and is transmitted through a conduit or gas inlet pipe 15 into a nozzle 12 through which flows the reductant metal, such as sodium. Sodium is provided from the head tank 110 or supply vessel 105. As is understood, these vessels may be combined into one or may be several, it being within the

skill of the art to design the exact combination of parts in the system. Liquid metal pump 105 provides a continuous flow of liquid metal to the nozzle 12 and the amount of liquid metal and gas is adjusted to maintain the temperature in the reactor 11 at a predetermined but generally low temperature of about 400°C. It is understood that various temperatures may be selected as the operating temperature but about 400°C is preferred at the present time. The reaction products in the reactor 11, as previously explained in the Armstrong et al. patents incorporated herein by reference and the illustrated example herein, comprise a slurry of excess sodium, sodium chloride particles and titanium particles. This slurry flows through outlet conduit 18 into the receiving vessel 20. The temperature of the material at this time is still approximately the outlet temperature from the reactor 11 which may be for instance, about 400°C. In the vessel 20, the slurry is stirred in the vessel when the agitator 32 is operated by virtue of the motor 30. The slurry exits the vessel 20 through the discharge outlet 25 thereof into the indexing filter system 35.

Through gravity, the slurry entering the indexing filter system 35 flows into the T-connector 66 and then into inlet conduit 63 through the filter 60 and into the outlet conduit 65. As the liquid sodium flows through the filter 60 which may be for instance, a 125 micron wedge wire filter, the solids concentration increases as liquid sodium drains. The compaction ram assembly 70 is actuated and the piston 72 drives forwardly into the chamber 56 compressing the material in the conduit 66 thereby expressing liquid metal through the filter 60 until a cake is formed in which most of the liquid metal has been expressed and there remains what could be categorized as wet cake particulate salt and particulate titanium. This cake has sufficient integrity to hold its shape but at the same time still contains some liquid metal. As indicated in the drawing, liquid metal which exits the indexing filter system 35 through the outlet conduit 65 is then recycled to the head tank 110 and moved via the pump

108 back to the nozzle 12 in the reactor 11. After compression by the compaction ram 70 is complete, the motor therefor 74 withdraws the piston 72 and the indexing disc 55 is rotated by the index drive mechanism 45 so as to advance the next chamber 56 into position for another actuation. As is seen from the drawing, because the inlet conduit 66 is connected via gravity to the vessel 20, as soon as the compaction ram 70 withdraws more slurry enters the system. As the disc 55 is rotated as soon as the ram is withdrawn, no slurry material enters the chamber 56 after compaction until the next chamber is in alignment with the compaction ram 70, at which time the aligned chamber 56 fills with the slurry and is thereafter compacted or compressed.

The discharge ram assembly 80 is in alignment or in registry with another chamber 56 of the indexing disc 55 and when the chamber 56 which has the compressed or compacted material therein is in alignment with the discharge ram 80 the piston 82 moves the cake in the chamber 56 into the distillation system 95.

As seen from Fig. 3, there are a plurality of seals 61 and 86 which contain the liquid metal in the appropriate conduit and in its restricted path. However, seals are not necessarily perfect in the real world and although the sealing mechanism in the conduits are intended to provide a seal for the liquid metal, some inevitably may escape and is collected within the housing 36 and flows out of the outlet 43 and conduit 44 into the condenser assembly 100 for further recycle, as will be explained. Although illustrated with one compaction ram assembly 70 and one discharge ram assembly 80, it is well within the skill of the art to include more than one compaction and/or discharge ram assembly 70, 80.

In the distillation system 95, the particulates from the cake are heated by virtue of the heat exchanger 97, which may be via conduction, convection, induction heating or any other suitable commercial method of heating the powder or particulates moved by the conveyor 96

through the cake breaker 93 to the vessel 120. The cake breaker 93 is shown schematically and may be a fixed series of wires or a variety of other mechanical mechanisms which breaks a compacted particulate matter into a loose friable material. Liquid metal which is vaporized in the distillation system 95 is collection and transmitted via conduits 101 into the condenser assembly 100 and container 103 which is maintained at a sufficiently low temperature to condense the liquid metal vapor into a liquid which is transmitted to storage, such as in head tank 110. As previously described, eventually the liquid metal in the tanks 105 and 110 is recirculated via the pump 108 to the reactor 11 and more particularly the nozzle 12. A valve 121 exists between the vessel 120 and the vessel or lock hopper 125. In addition, pump 122 is in communication with the vessel or lock hopper 125 to ensure no vapors back-up into the system 10 and to evacuate the system if it is necessary to isolate the lock hopper 125 by actuation of the valves 121 and 126. From the lock hopper 125 granular material is transmitted to the passivation system 130 and more particularly to a conveyor 132 which moves within a containment vessel 131 while a passivating gas or liquid is provided through the gas or water inlet or conduit 133. As illustrated, the particulates move in countercurrent relation to the passivation material but may not be required to do so. Preferably, the passivation fluid is a gas containing a small percentage such as 0.2% by volume oxygen and an inert gas such as argon. The passivated material then moves through outlet 136 to a wash and dry system 140. A pump 135 and conduit 134 exhaust the passivating fluid and recycle same, if desired.

Referring now to Fig. 5, there is shown an alternate embodiment of the present invention in which like numbers have been applied to like parts. The principal difference in the embodiment of Fig. 5 is that the indexing disc 55 as well as the indexing filter system 35 is arranged horizontally instead of vertically so that as slurry drains from the bottom of the

vessel 20, liquid reducing metal drains through the filter 60 and a compaction occurs after rotation of the indexing disc 55. Thereafter upon further rotation, the discharge ram assembly 80 is actuated to move the cake into the distillation system 95. Other than the positioning of the indexing disc 55, requiring an indexing between recovering slurry and compaction , the operation of the two systems is identical.

On the assumption that a single Armstrong reactor can produce 2,000,000 pounds per year of product, such as titanium or a titanium alloy such as titanium, 6% aluminum and 4% vanadium, the chambers 56 would be 10 inches in diameter and 6 inches in length. Preferably there are 6 such chambers in each disc 55 based calculations that the slurry and/or gel produced in the Armstrong Process which is approximately 22-23% solids by weight. The indexing disc 55 will be indexed approximately every 11 seconds based on the above-described chambers. Different volume chambers or number of chambers will require different indexing times, but this is well within the skill of the art. Under the present example, the material in the chamber 56 will be compressed by a factor of 4 so that the cake ejected by the compaction ram assembly 80 will be about 1.5 inches thick and have a solids composition between about 64 and 65 percent by weight.

Referring now to Fig. 6, there is disclosed a schematic representation of the various processes and products made in accordance with the present invention. The reduction box is the Armstrong Process in which a exothermic reduction occurs controlled by the use of constituents taught in the above-mentioned and incorporated Armstrong et al patents. The separation is as previously described herein along with the passivation. The passivated material then is transmitted to a wash and dry assembly 140 in which the salt product, in this specific example, sodium chloride, is removed from the product particulates, in the example titanium or titanium alloy powder. Referring to Fig. 6, the schematic shows that the powder

may be melted to form an ingot or other solid product by a variety of methods such as casting or transmitted to a powder metallurgy process which includes, but is not limited to, for instance isostatic cold processing, hot isostatic processing using pressure to densify the metal powder into a predetermined shape and density. The product may also be produced by cold spraying the metal powder in a gas jet or subjecting the metal powder to a laser or spheridizing the metal powder by plasma. The metal powder may be formed into a foam and thereafter pressed and sintered to form a stable metal foam as is well known in the art. The powder may be pressed onto a mandrel and thereafter rolled into a thin wall tube. Moreover, powder product may be formed by drawing or extruding the metal powder. In the event that product morphology such as the packing fraction, mean size or size distribution needs to be altered, attriting mechanism may be used to change the morphology of the powder, including the packing fraction or reducing the overall size distribution of the powder.

All these methods of producing product as well as the products formed thereby when combined with the present separation process are included in the present invention.

P-trap is the pressure above the filter (assume downstream pressure remains constant) as the run progressed. Flow 2 is the Na flow rate and the V reactor shows when the product was made. At t=8420, sodium flow was initiated to the trap. Trap pressure remained relatively constant as the Na flowed through the clean filter (125 micron) until the reactor valve was opened and started to build cake. The cake DP grew in a linear fashion until t=8520 when the reaction rate began to slow because of nozzle plugging due to subsonic operation of the nozzle. The cake thickness after distillation was measured to be on average 5 to 6 inches. The bottom of the cake appeared less dense than the top of the cake and measurements of the cake density showed a density in the top of the cake of 1.1 g/cc and in the bottom of the cake .73 g/cc. It is believed that the bottom was less dense because it was

formed at a lower pressure. For example, the DP is determined by the flow rate; for this run the flow rate was 30 kg/min. Also, after product production was terminated and Na flow continued, the cake appeared to compact further (see pressure increase while flow decreased after t=8550). Prior to Na flow shutdown, DP was up to 22 psig versus 18 psig when significant product production ended, see Figs. 1 and 2, see Figs. 7 and 8.

Heat was applied to the cake area and vapor was removed to a primary condenser out the top side of the trap and to a secondary condenser by distilling through the wedge wire filter. During the distillation, a total of 5.9 kg of Na was removed from the cake which weighed 3.4 kg after the distill. 3.8Kg of the 5.9 kg was found to have condensed in the secondary condenser, see Fig. 9.

In another nutsche run, the trap was designed to allow distillation through the filter into the bottom of the trap to utilize the full trap diameter for vapor movement. The trap also had the standard 1" line to a primary condenser, see Fig. 10. Heat was concentrated on the cake area while the bottom of the trap was maintained cool to support condensation of the Na. After distillation, 1.6 kg of Na went to the primary condenser and 1.3 kg. of Na distilled into the bottom of the trap leaving a 3.1 kg. cake of titanium and NaCl.

However, it has been found that breaking the filter cake drastically reduces the distillation times and rates for the distillation of the liquid metal, such as sodium. Using a breaker bar or some other mechanical means such as moving fingers or a mixer has significantly reduced the first portion of the vacuum distill from 40,000 - 50,000 seconds (11-14 hours) to 20,000 to 30,000 (between about 6 and 8 hours). The second portion of the distill, that is the decreasing temperature and pressure portion referred to as the tail was not affected by breaking the filter cake.

It has also been discovered that using a sweep of inert gas such as argon heated, preferably in the range of from about 500°C to about 800°C. during the second distill or tail portion reduced the amount of time necessary to distill the reductant metal (sodium) from about 40,000 - 50,000 seconds to about 10,000 seconds (about 3 hours.). This is a significant improvement over the prior method. By using either one of the methods or a combination of breaking the filter cake combined with an inert gas sweep, the distillation times can be decreased from about (22 or 28) hours to about (9 to 11) hours. This is of significant importance in the design of plants by simplifying designs, reducing collection tanks, valves, piping and other associated equipment.

After vacuum distillation is apparently complete, any remaining trapped reductant metal (sodium) becomes impractical to remove. While it seems obvious to introduce the filter cake into water to wash the residual salt (NaCl) from the titanium powder, the problem exists of trapped reductant metal (sodium) in the filter cake which when combined with water could produce a significant explosion. It is a fact that the mixture of sodium liquid and water will provide an explosion having energy greater than the equivalent amount of TNT.

It has been found in the production of Ti by the subsurface reduction of $TiCl_4$ by Na that crumbling the filter cake into small quantities, such as less than about five centimeters in diameter and preferably in the range of from about two to about five centimeters in diameter, during or subsequent to the distillation of sodium apparently makes particles or clumps small enough that any trapped Na is manageable without significant damage to equipment or harm to personnel, if proper care is taken in equipment design and with appropriate safety precautions. After distillation, the filter cake is friable and easily crumbled. To the extent that large quantities of crumbled filter cake can be water washed without fear of explosion significantly reduces the distillation times required in the production of the various elemental

material and alloys described in the above-referenced patents, particularly where sodium or other alkaline metal is used as a reductant.

Alternatively, it has been found that the entire distillation can be accomplished at positive pressure, such as, but not limited to, psig with a heated or hot inert gas, such as but not limited to Ar at about 500°C to about 800°C followed by cooling to condense the vaporized liquid metal, such as but not limited to Na. Thereafter, the cooled liquid metal will be returned for additional use.

Summarizing this invention relates to mechanism and methods for decreasing the distillation time of a filter cake produced by the process described in the above-referenced patents. The filter cake can be broken such as by vibration or moving mechanism in the filter cake area or by stationery mechanical bars or members in the filter cake area or other suitable mechanism. An inert sweep gas with or without vacuum can be used alone or in combination with the above described methods breaking the filter cake during the distillation in order significantly to reduce the distillation time of the liquid metal in the filter cake.

Referring to Fig. 11 of the drawings, there is shown a transfer mechanism 10E which includes a double walled conduit including an outer conduit wall 11 having a liquid outlet 12 and end walls 13, the wall 11 being preferably but not necessarily cylindrical. Interior of the cylindrical wall 11 is an inner tube or conduit 15 having a portion 16 which is solid and a portion 17 which is apertured and may be a mesh of any suitable size. The inner tube or conduit 15 may either be cylindrical as illustrated in Fig. 1 or conical as will be explained, the inner conduit 15 has a discharge end 18 thereof which opens into a vacuum chamber 25 and has an inlet end 19 thereof which opens into a container or vessel 20 in communication with the reactor as illustrated in the Armstrong patents previously referenced and incorporated herein.

A feed screw 30 is positioned within the inner conduit 15 and includes a rotatable shank 31 having a conical thread 32 positioned on the shank 31 as is well known in the art. The thread 32 may have a constant or a variable pitch. The pitch is the distance between adjacent threads and the variable pitch may preferably be a progressive pitch in which the pitch decreases from the vessel 20 toward the container or vessel 25, for a purpose hereinafter described.

In the preferred but not limiting embodiment of the present invention, the transfer mechanism 10E is used in conjunction with a material made by the Armstrong Process. More particularly, for purposes of illustration only, the slurry discussed herein will be a combination of liquid sodium, sodium chloride particles and particles of titanium and/or a titanium alloy. As set forth in the Armstrong patents, a variety of metal and non-metal products may be made thereby and it is intended that the present invention not be limited to any particular product made by the Armstrong Process and certainly not limited to the preferred product described herein.

In any event, the vessel or container 20 preferably operated under an inert atmosphere or under vacuum has therein a slurry of the particles previously described and as the slurry enters the portion 19 of the inner conduit or tube 15 and the feed screw 30 is rotated as illustrated in the drawings by rotation of the shank 31, the slurry is moved along the feed screw from left to right as illustrated in Fig. 11. Because of the progressive pitch of the feed screw 30 in Fig. 11, that is the threads 32 thereof are closer together so that the pitch decreases from left to right, the material is concentrated as it is moved from the container or vessel 20 to the container or vessel 25. Moreover, because the portion 17 of the conduit or tube 15 is apertured or porous, liquid sodium drains therethrough and passes out of the outlet 12 for further processing. Therefore, the slurry as it is transported from container or vessel

20 to container or vessel 25 becomes more concentrated as liquid is drained therefrom and the density increases as the pitch between the adjacent threads diminishes.

Another way to express what occurs is that the volume between adjacent threads and the wall of the cylinder or tube 16 diminishes as material is moved by the feed screw 30 from container or vessel 20 to container or vessel 25. By the time the slurry is concentrated and reaches the portion 16, the solid portion 16 of the inner tube or conduit 15, a seal is established between the vessel 25 and the vessel 20 which houses the slurry from the reactor. The formation of a seal by the transfer mechanism 10 is a critical aspect of the present invention because separation of liquid sodium and salt from the desired particles of the ceramic or metal alloy, as described in the Armstrong patents may include distillation in a vacuum chamber or a vessel 25 and the Armstrong reactor itself may be an inerted vessel such as with argon. Accordingly, it is important for a seal to be formed between the two containers or vessels in order to permit continuous operation between the two vessels without the necessity of shutting down one of the vessels during transfer or destroying the protective atmosphere in the vessel 20 or the vacuum in vessel 25.

Referring to Figs. 12 and 13, there are disclosed alternate embodiments of the invention. Again with the principle feature that the volume between adjacent screw threads and the container or housing in which the feed screw is positioned diminishes from vessel 20A to vessel 25A. As seen in Fig. 12, the transfer mechanism 10F has a housing 15A conical in shape and the screw 30 therein may or may not be a progressive pitch screw. The screw threads in the embodiment illustrated in Fig. 12 may not need to be closer together, that is the pitch need not be diminished in order to reduce the volume of the material between adjacent threads and the housing wall as the material is moved from left to right or from vessel 20A to vessel 25A. However, it may be advantageous to use both the conical shaped

inner housing 15A with or without a progressive screw 30A depending on engineering considerations.

Referring to Fig. 13, there is shown another embodiment of the present invention 10G in which the shank 31B of the screw 30B is conical in shape with the larger end of the cone being adjacent the vessel 25B and with the pitch between adjacent threads 32B being constant or diminishing. In either case, the volume of the area between adjacent threads and the inner container 15B diminishes as the material is moved from the vessel 20B to the vessel 25B.

Although the invention has been described with respect to an inerted vessel and a vacuum vessel, the invention includes movement and concentration of material from one container to another without compromising the environment of either container. The containers may be connected pipes or vessels, and the environments may be vacuums, inerted atmospheres or otherwise. Central to the invention is concentration of solids in a slurry to transport solids from one environment to another while forming a seal therebetween so as to isolate the environments from each other.

Referring to Fig. 14, there is shown a separation schematic which is a combination of the above referenced applications illustrating another separation process, system and method relating to but not limited by the Armstrong Process, previously described. As in the earlier described systems, apparatus and methods, there are supplies of halide(s) and reductant metal(s) introduced as previously shown into a reactor to produce by exothermic reaction, in one instance, a slurry of a metal powder, salt particles, and excess reductant metal. The reactor is operated under an inert atmosphere, as taught in the Armstrong Patents, and the slurry produced is transferred to a first vessel, also operated under an inert atmosphere and /or vacuum, as will be explained since the separation process may include portions thereof at either or both positive or negative pressure, that is the first vessel is inerted. It is very

important to prevent oxygen contamination of the first vessel during the processing of the slurry to allow continuous production without shutting down the first vessel and seasoning with liquid metal or otherwise to remove oxygen contamination.

There are a variety of means by which the slurry is treated to remove unwanted constituents, such as salt particles and excess reductant metal. But, all involve moving excess reductant metal as liquid or vapor or both from the first vessel, leaving either a wet or dry cake of either metal powder or a combination of metal powder and salt particles. In order to preserve the integrity of the first vessel, there is a second vessel which may be a tank or a pipe or any container operated under an inert atmosphere and/or vacuum into which the treated slurry(either wet or dry and either metal powder or a combination of metal powder and salt particles) is transferred for further processing and which has some lock or valve or seal or plug mechanism to enable transfer of product from the inerted vessels or containers to a non-inerted environment. As seen in the drawings as previously described herein, particularly Fig.1, there is shown an apparatus in which the slurry is heated in the first vessel to remove excess liquid metal as both liquid and as vapor. The dried cake is transferred to a second inerted vessel for commutation. Agitation in the first vessel, by mechanical means or otherwise speeds up the distillation process and simultaneously breaks the filter cake when formed, also an added benefit. However, without much change as illustrated in Fig.14, the cake can both be broken and the excess liquid reductant metal removed by an inert hot sweep gas, which if hot enough will vaporize not only the excess liquid reductant metal but also the salt particles, leaving the metal powder to move into the second container(which may be a pipe) to pass through the lock mechanism into an optional passivation station or to be packaged without passivation in an inert environment for shipping. As seen, therefore, the first vessel can be used to treat the slurry by heating to produce either metal powder alone or

in combination with salt particles.

As seen in Figs. 2-6, the first vessel in system 10A can include a sequential indexing separation system in which the first vessel of Fig.14 is a combination of receiving vessel 20 and indexing filter system 35 which produces a sequence of squeezed cakes to a second vessel or distillation system 95, as previously described. The second vessel or container of Fig.14 may be disposed horizontally as the distillation system 95 in system 10A illustrated in Figs. 2-6 and may or may not have a conveyor therein, all depending on engineering considerations within the skill of the art. As seen in Fig. 14, material in the second container, which is now dry, but may be either metal powder alone or a combination of metal powder and salt, is moved one way or another through a lock, as illustrated, or a seal or a valve or any comparable mechanism from the inerted condition of the second container to another environment for additional treatment, which may include passivation followed by water washing or simply packaging in inerted shipping containers, as necessary. The lock mechanism may be the variable pitch screw of Fig. 11 or the variations thereof in Figs. 12 and 13 or any other suitable lock mechanism, such as but not limited to a gate valve.

As seen, therefore, the treatment of the slurry in the first vessel under an inert atmosphere or vacuum or combinations thereof may produce a wet or dry cake of metal powder or a combination of metal powder and salt, depending on the separation conditions chosen. Heaters, either internal or external or both, and/or hot inert sweep gases may be used at either positive or negative pressures or combinations of both positive and negative pressures with either the heaters or sweep gas or both to move material, either excess reductant metal alone or a combination of reductant metal and salt, out of the first vessel. Depending on what is transferred from the first vessel, the second container may employ a hot inert sweep gas or other mechanism to isolate the metal powder from unwanted constituents.

Thereafter, transfer is made to the conveyor, or alternate mechanism, for additional treatment or handling. Passivation with an inert gas having a small amount of oxygen after cooling may be employed, as previously described, followed by a water wash and drying before packaging. Alternately, if both the reductant metal and the salt are removed in the first vessel and/or the second container, the water washing and/or passivation may not be required, resulting perhaps in lower oxygen contamination and/or expense.

While there has been disclosed what is considered to be the preferred embodiment of the present invention, it is understood that various changes in the details may be made without departing from the spirit, or sacrificing any of the advantages of the present invention.

WE CLAIM:

1. A method of separating metal powder from a slurry of liquid metal and metal powder and salt, comprising introducing the slurry into a first vessel operated in an inert and/or vacuum environment for separation of liquid metal from the metal powder and salt leaving principally salt and metal powder substantially free of liquid metal, transferring the salt and metal powder substantially free of liquid metal to a second vessel operated in an inert environment, and thereafter treating the salt and metal powder to produce passivated metal powder substantially free of salt and liquid metal.

2. The method of claim 1, wherein the inert environment is an argon atmosphere.

3. The method of claim 1, wherein the salt and metal powder are crushed to form clumps having diameters less than about five centimeters prior to passivation.

4. The method of claim 1, wherein the liquid metal is separated from the salt and metal powder in the first vessel both as a liquid and as a vapor.

5. The method of claim 4, wherein the liquid metal vapor from the first vessel is transferred to a condenser operated in an inert environment.

6. The method of claim 4, wherein the liquid metal is an alkali or an alkaline earth metal or mixtures thereof.

7. The method of claim 6, wherein the salt is a halide.

8. The method of claim 7, wherein the metal powder is titanium or a titanium alloy.

9. The method of claim 8, wherein the titanium or titanium alloy is CP 1 to CP 4.

10. The method of claim 9, wherein the metal powder has diameters in the range of from about 0.1 to about 10 microns.

11. The method of claim 1, wherein passivation occurs on a conveyor.

12. The method of claim 11, wherein the metal powder is continuously cooled and passivated.

13. The method of claim 1, wherein the environments of the first and second vessels are protected from contamination by oxygen during the production of metal powder substantially free of salt and liquid metal.

14. A method of separating metal powder from a slurry of liquid metal and metal powder and salt formed by introducing a metal halide vapor subsurface of a liquid metal causing an exothermic reaction producing salt and metal powder with the liquid metal being present in excess of the stoichiometric amount required, comprising introducing the slurry into a first vessel operated in an inert and/or vacuum environment for filtration and vaporization of liquid metal from the metal powder and salt leaving principally salt and metal powder substantially free of liquid metal, transferring the liquid metal vapor to a condenser operated in an inert environment to convert the liquid metal vapor to a liquid to be recycled for production of additional metal powder, transferring the salt and metal powder substantially free of liquid metal to a second vessel operated in an inert environment, and thereafter treating the salt and metal powder to produce passivated metal powder substantially free of salt and liquid metal.

15. The method of claim 14, wherein the slurry is heated in the first vessel by contact with a heat exchanger internal to the first vessel having heat exchange fluid pumped therethrough.

16. The method of claim 14, wherein the liquid metal vapor from the first vessel is cooled by contact with heat exchanger internal to the condenser having a heat exchange fluid pumped therethrough.

17. The method of claim 14, wherein the first vessel is heated by both an internal and an external heat exchanger.

18. The method of claim 14, wherein the slurry is introduced into the interior of a candle filter in the first vessel with liquid metal flowing through the candle filter and out of the first vessel.

19. The method of claim 14, wherein the inert environment for the first and second vessels is an argon atmosphere.

20. The method of claim 19, wherein the condenser is operated in an argon atmosphere.

21. The method of claim 14, wherein the environments of the first and second vessels are protected from contamination by oxygen during the production of metal powder substantially free of salt and liquid metal.

22. A system for separating metal powder from a slurry of liquid metal and metal powder and salt formed by introducing a metal halide vapor subsurface of a liquid metal causing an exothermic reaction producing salt and metal powder with the liquid metal being present in excess of the stoichiometric amount required, comprising a first inerted vessel in communication with a heater and a filter for filtering liquid metal from the slurry and for heating liquid metal to vaporize the liquid metal from the salt and metal powder forming a filter cake of salt and metal powder, an inerted condenser in communication with said first vessel for receiving metal vapor and converting same to liquid metal, a second inerted vessel in valved communication with said first inerted vessel for receiving filter cake therefrom; a crusher in or in communication with said second inerted vessel for crushing the filter cake; a cooling and passivating station for receiving crushed filter cake, and a valve mechanism intermediate said first and second vessel and between said second vessel and said cooling and

passivating station to prevent air from contaminating said first and second vessels during transfer of filter cake from said first vessel to said cooling and passivating station.

23. The system of claim 22, wherein said heater in communication with said first inerted vessel is interior of said vessel.

24. The system of claim 23, wherein said heater interior of said inerted first vessel is in communication with a source of heat exchange fluid which optionally is dedicated to said heater.

25. The system of claim 22, wherein said filter in communication with said first inerted vessel is interior of said vessel.

26. The system of claim 25, wherein said filter is a filter forming an annulus with said first inerted vessel into which liquid metal flows, and further including a conduit in communication with said annulus for transferring liquid metal from said first inerted vessel to an inerted liquid metal reservoir.

27. The system of claim 22, wherein said first and second inerted vessels are inerted with argon.

28. The system of claim 27, wherein said condenser is inerted with argon.

29. The system of claim 28, wherein said inerted condenser is in communication with an argon inerted reservoir for liquid metal formed from condensed metal vapor.

30. The system of claim 22, wherein said condenser is in communication with a source of heat exchange fluid which optionally is dedicated to said condenser.

31. The system of claim 22, wherein said valve intermediate said first and second inerted vessel is hinged to open into said second inerted vessel.

32. The system of claim 22, wherein said first and second vessel are integral.

33. A method of separating metal particulates from a slurry of liquid metal and metal particulates and salt particulates, comprising filtering the slurry to form a cake of metal and salt particulates with some liquid metal, breaking the cake and removing liquid metal from the broken cake, and thereafter separating the metal and salt particulates.

34. The method of claim 33, wherein the liquid metal is removed from the broken cake by vacuum distillation.

35. The method of claim 33, wherein the liquid metal is removed from the broken cake with a hot sweep gas.

36. The method of claim 35, wherein the hot sweep gas is an inert gas.

37. The method of claim 36, wherein the inert gas is argon.

38. The method of claim 36, wherein the hot sweep gas is at positive pressure.

39. The method of claim 37, wherein the hot argon sweep gas is at positive pressure.

40. The method of claim 33, wherein the liquid metal is present in the filter cake up to about ten times the weight of the metal particulates.

41. The method of claim 33, wherein the liquid metal is an alkali metal or an alkaline earth metal or mixtures thereof.

42. The method of claim 33, wherein the liquid metal is Na or Mg.

43. The method of claim 33, wherein the metal particulates are Ti.

44. The method of claim 33, wherein the metal particulates are a Ti alloy.

45. The method of claim 33, wherein the salt particulates are a halide.

46. The method of claim 33, wherein the salt particulates are a chloride.

47. The method of claim 33, wherein the metal particulates are Ti or a Ti alloy and the salt is Na or Mg chloride.

48. The method of claim 47, wherein the liquid metal is Na and the salt particulates are NaCl.

49. The method of claim 33, wherein the cake is broken into pieces having diameters up to about five centimeters.

50. The method of claim 33, wherein the cake is broken into pieces having diameters up to about two centimeters.

51. A method of separating metal particulates from a slurry of liquid metal and metal particulates and salt particulates, comprising filtering the slurry to form a cake of metal and salt particulates with some liquid metal, breaking the cake and removing liquid metal from the broken cake, separating the metal and salt particulates, and sizing the metal particulates before water washing to prevent unacceptable explosions upon contact with water.

52. The method of claim 51, wherein the liquid metal is removed from the broken cake by vacuum distillation or by a hot sweep gas.

53. The method of claim 52, wherein the hot sweep gas is argon.

54. The method of claim 52, wherein the hot sweep gas is at positive pressure.

55. The method of claim 53, wherein the hot argon sweep gas is at positive pressure.

56. The method of claim 52, wherein the liquid metal is Na or Mg and is present in the filter cake up to about ten times the weight of metal particulates.

57. The method of claim 56, wherein the metal particulates are Ti or a Ti alloy.

58. The method of claim 57, wherein the cake is broken into pieces having diameters up to about five centimeters.

59. The method of claim 58, wherein the cake is broken into pieces having diameters up to about two centimeters.

60. A transfer mechanism between a first vessel containing a slurry of liquid and solids and a second vessel under vacuum, comprising a housing in communication with said first and said second vessels, a screw having a plurality of helical threads along a longitudinal shank within said housing for transferring material from said first vessel to said second vessel, the volume between adjacent screw threads and said housing diminishing between said first and said second vessels, whereby slurry entering said housing from said first vessel has the solids therein concentrated as the slurry is transported by said screw toward said second vessel while liquid is expressed from the slurry as the solids are concentrated until the concentrated solids form a plug sealing said second vessel from said first vessel.

61. The transfer mechanism of claim 60, wherein said screw is a variable pitch screw.

62. The transfer mechanism of claim 60, wherein said screw is a progressive pitch screw with the smallest pitch being nearest said second vessel.

63. The transfer mechanism of claim 60, wherein said housing is generally cylindrical.

64. The transfer mechanism of claim 60, wherein said housing is conical with the smallest end being nearest said second vessel.

65. The transfer mechanism of claim 60, and further including in said transfer mechanism slurry of liquid metal and salt particles and particles of a ceramic or a metal or an alloy.

66. The transfer mechanism of claim 65, wherein said liquid metal is Na or Mg.

67. The transfer mechanism of claim 66, wherein said particles of a ceramic or a metal or an alloy are Ti or an alloy thereof.

68. The transfer mechanism of claim 60, wherein said housing is cylindrical and said screw is a progressive pitch screw with the smallest pitch being nearest said second vessel.

69. The transfer mechanism of claim 60, wherein said housing is conical with the smallest end being nearest said second vessel and said screw has threads of constant pitch.

70. The transfer mechanism of claim 60, wherein said shank has an increasing diameter toward said second vessel.

71. The transfer mechanism of claim 60, wherein at least a part of said housing in liquid communication with said first vessel has a plurality of apertures therein.

72. The transfer mechanism of claim 60, wherein the plurality of apertures is a mesh.

73. The transfer mechanism of claim 60, and further comprising an outlet in said housing for separating liquid flowing through said apertures from the slurry.

74. A transfer mechanism between a first vessel containing a slurry of liquid alkali or alkaline earth metal or mixtures thereof and metal or alloy or ceramic particles and halide salt particles and a second vessel under vacuum, comprising a housing in communication with said first and said second vessels, a screw having a plurality of helical threads along a longitudinal shank within said housing for transferring material from said first vessel to said second vessel, the volume between adjacent screw threads and said housing diminishing between said first and said second vessels, whereby slurry entering said housing from said first vessel has the particles therein concentrated as the slurry is transported by said screw toward said second vessel while liquid metal is expressed from the slurry as the particles are

concentrated until the concentrated particles form a plug sealing said second vessel from said first vessel.

75. The transfer mechanism of claim 74, wherein said screw is a progressive pitch screw with the smallest pitch being nearest said second vessel.

76. The transfer mechanism of claim 74, wherein said housing is generally cylindrical.

77. The transfer mechanism of claim 74, wherein said housing is conical with the smallest end being nearest said second vessel.

78. The transfer mechanism of claim 74, wherein said housing is cylindrical and said screw is a progressive pitch screw with the smallest pitch being nearest said second vessel.

79. The transfer mechanism of claim 74, wherein said housing is conical with the smallest end being nearest said second vessel and said screw has threads of constant pitch.

80. The transfer mechanism of claim 79, wherein said shank has an increasing diameter toward said second vessel.

81. The transfer mechanism of claim 74, wherein at least a part of said housing in liquid communication with said first vessel has a plurality of apertures therein.

82. The transfer mechanism of claim 81, wherein the plurality of apertures is a mesh.

83. The transfer mechanism of claim 74, and further comprising an outlet in said housing for separating liquid flowing through said apertures from the slurry.

84. The transfer mechanism of claim 83, and further including a slurry of liquid Na, particles of NaCl and particles of Ti or an alloy thereof.

85. The transfer mechanism of claim 83, wherein a double wall housing is provided wherein the inner wall has a portion thereof apertured and a portion thereof solid and the outer wall has said outlet therein, said screw being positioned within said inner wall.

86. A method of concentrating and transferring a slurry from one container to another while sealing the containers, comprising providing communication between the containers, transporting slurry from one container toward another container while expressing liquid from the slurry thereby increasing the solids concentration thereof until a plug is formed between two containers while solids from the plug are transferred to the another container.

87. The method of claim 86, wherein one container is operated under an inert atmosphere.

88. The method of claim 86, wherein one container is operated under vacuum.

89. The method of claim 86, wherein the slurry contains liquid metal and metal particles.

90. The method of claim 89, wherein the slurry contains liquid alkali or alkaline earth metal.

91. The method of claim 86, wherein slurry contains liquid sodium metal and particles of Ti or an alloy thereof.

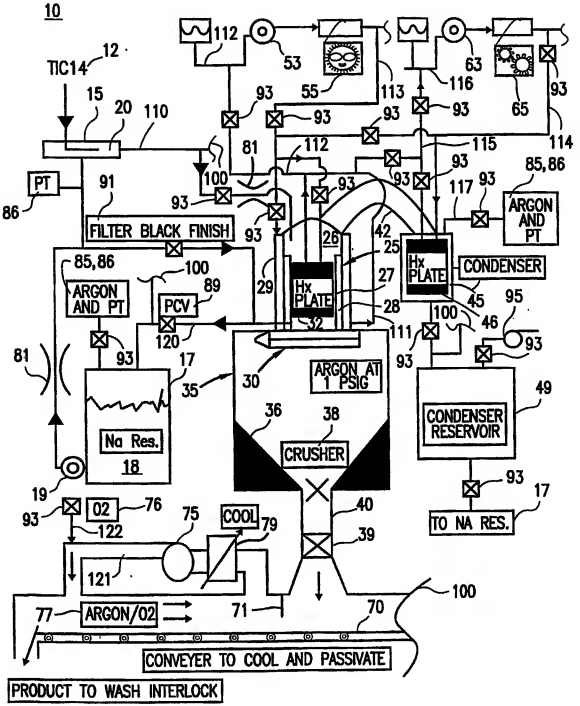
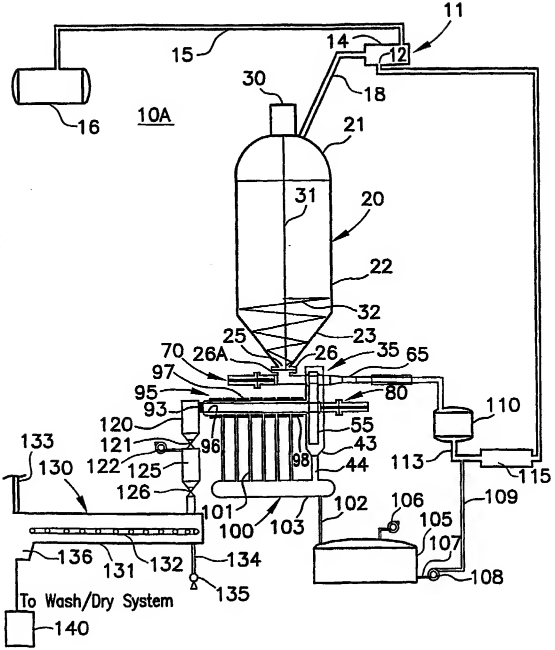

10
TIC14
12
15
20
110
112
53
55
113
116
63
65
93
114
115
81
100
91
PT
86
ARGON AND PT
85,86
FILTER BLACK FINISH
26
42
25
Hx PLATE
27
28
29
32
CONDENSER
45
46
95
89
PCV
120
17
35
30
111
ARGON AT 1 PSIG
36
38
CRUSHER
Na Res.
18
19
49
CONDENSER RESERVOIR
TO NA RES.
02
76
75
COOL
79
40
39
122
121
ARGON/02
77
71
70
CONVEYER TO COOL AND PASSIVATE
PRODUCT TO WASH INTERLOCK

FIG. 1



11
14
12
15
30
18
16
10A
21
31
20
22
32
25
23
70
26A
26
35
65
97
95
80
93
110
120
55
133
121
96
43
130
122
125
98
44
113
115
126
101
102
106
109
100
103
105
107
136
131
132
134
108
135
To Wash/Dry System
140

FIG. 2

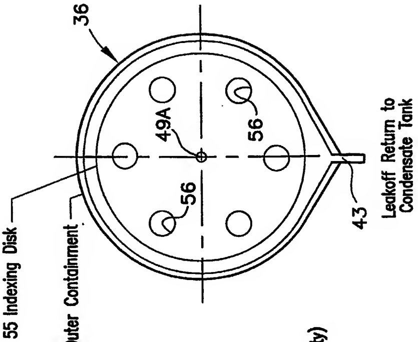

36
55 Indexing Disk
38 Outer Containment
49A
56
56
43
Leakoff Return to Condensate Tank

FIG. 4

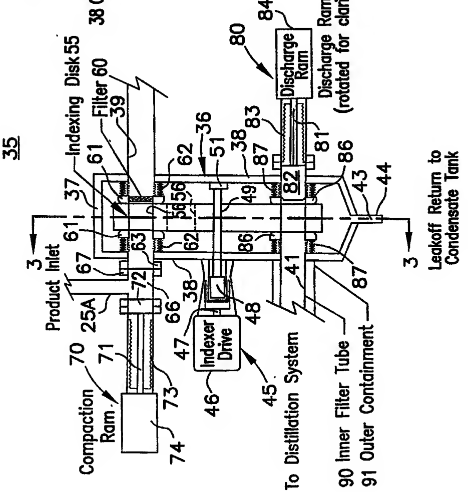

35
3
Product Inlet
25A
67
61
63
72
66
37
Indexing Disk 55
Filter 60
39
61
62
56
56
62
36
51
38
38
49
87
86
86
82
87
83
81
80
84
Discharge Ram
Discharge Ram (rotated for clarity)
41
43
44
Leakoff Return to Condensate Tank
3
87
70
71
73
74
Compaction Ram
47
48
Indexer Drive
46
45
To Distillation System
90 Inner Filter Tube
91 Outer Containment

FIG. 3

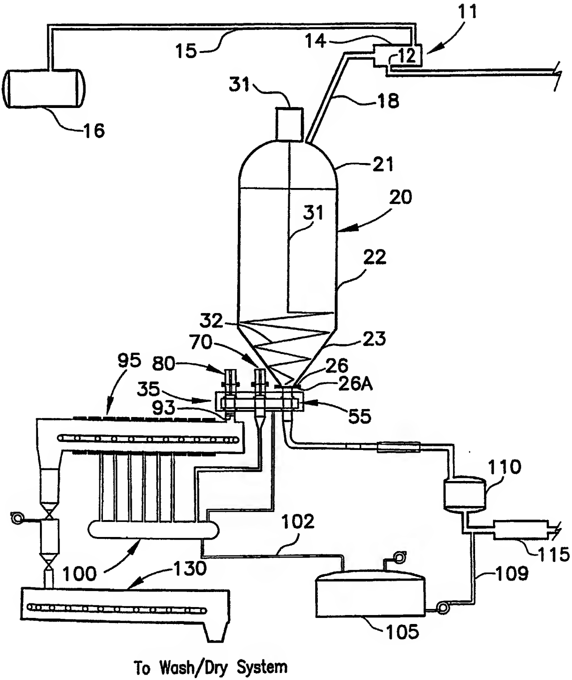

11
14
12
15
16
31
18
21
20
31
22
32
70
23
95
80
26
26A
35
93
55
110
102
115
100
130
109
105
To Wash/Dry System

FIG. 5

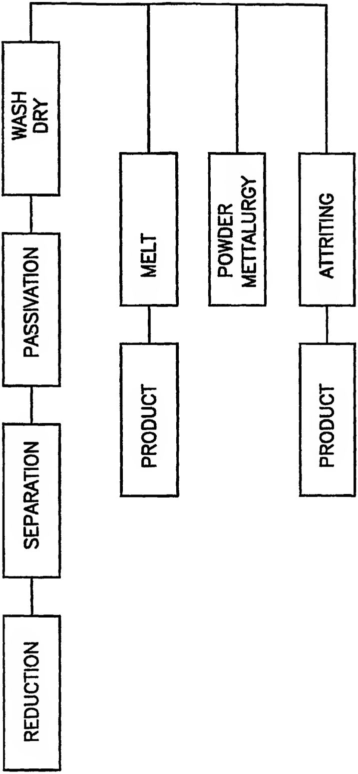

REDUCTION
SEPARATION
PASSIVATION
WASH
DRY
MELT
PRODUCT
POWDER
METTALURGY
ATTRITING
PRODUCT

FIG. 6

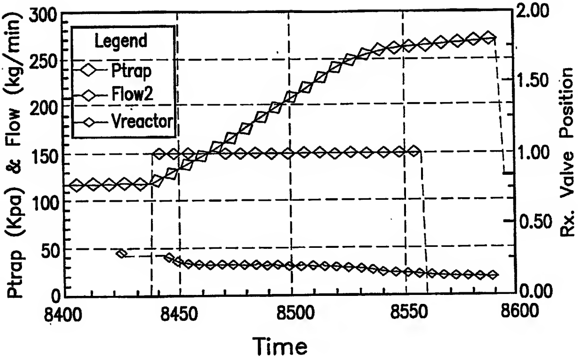

Legend
Ptrap
Flow2
Vreactor
Ptrap (Kpa) & Flow (kg/min)
300
250
200
150
100
50
0
Rx. Valve Position
2.00
1.50
1.00
0.50
0.00
8400
8450
8500
8550
8600
Time

FIG. 7

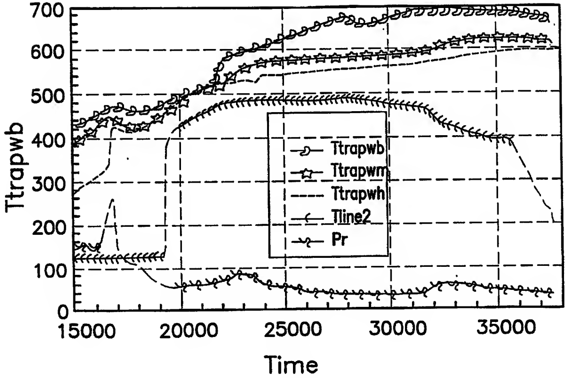

700
600
500
400
300
200
100
0
15000
20000
25000
30000
35000
Time
Ttrapwb
Ttrapwb
Ttrapwm
Ttrapwh
Tline2
Pr

FIG.8

The distillation setup for this trap was as follows:

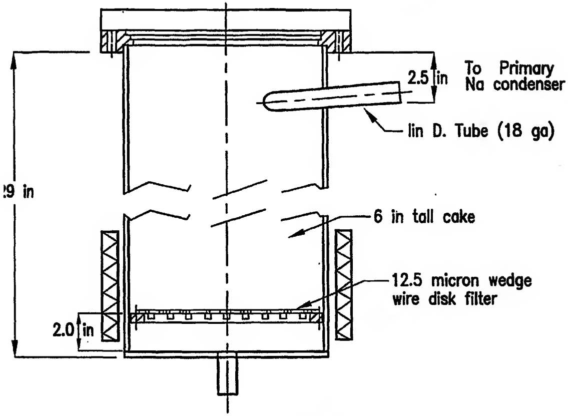


FIG.9

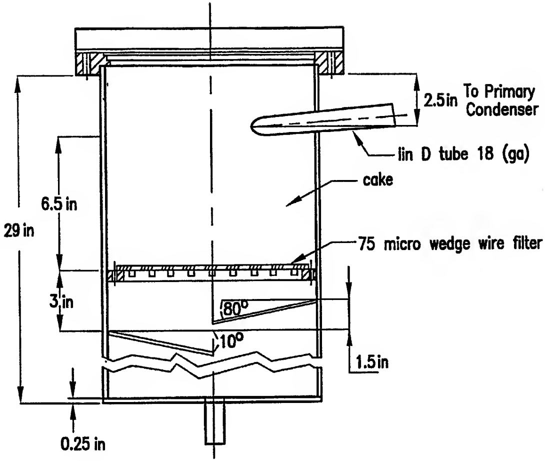

2.5in
To Primary
Condenser
lin D tube 18 (ga)
cake
6.5 in
29 in
75 micro wedge wire filter
3 in
80°
10°
1.5in
0.25 in

FIG.10

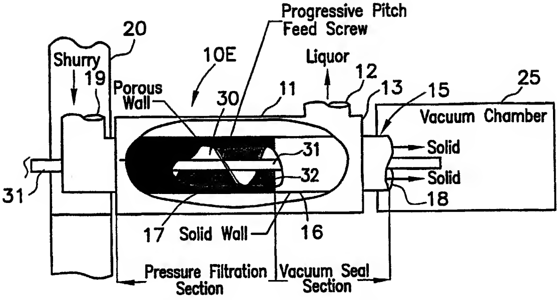

Progressive Pitch Feed Screw
20
Shurry
19
10E
Liquor
12
13
15
25
Porous Wall
30
11
Vacuum Chamber
Solid
31
32
Solid
31
18
17
Solid Wall
16
Pressure Filtration Section
Vacuum Seal Section

FIG.11

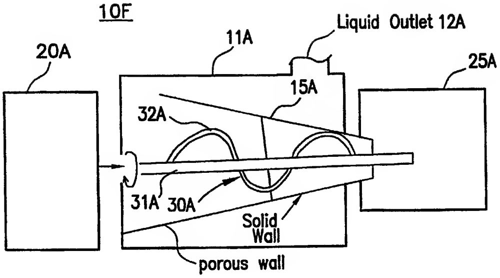

10F
Liquid Outlet 12A
11A
20A
25A
15A
32A
31A
30A
Solid Wall
porous wall

FIG. 12

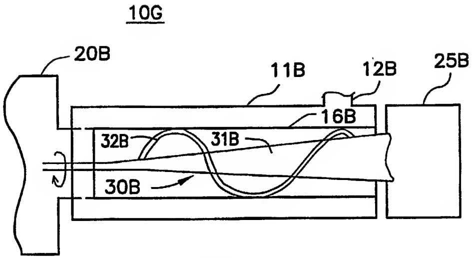


FIG.13

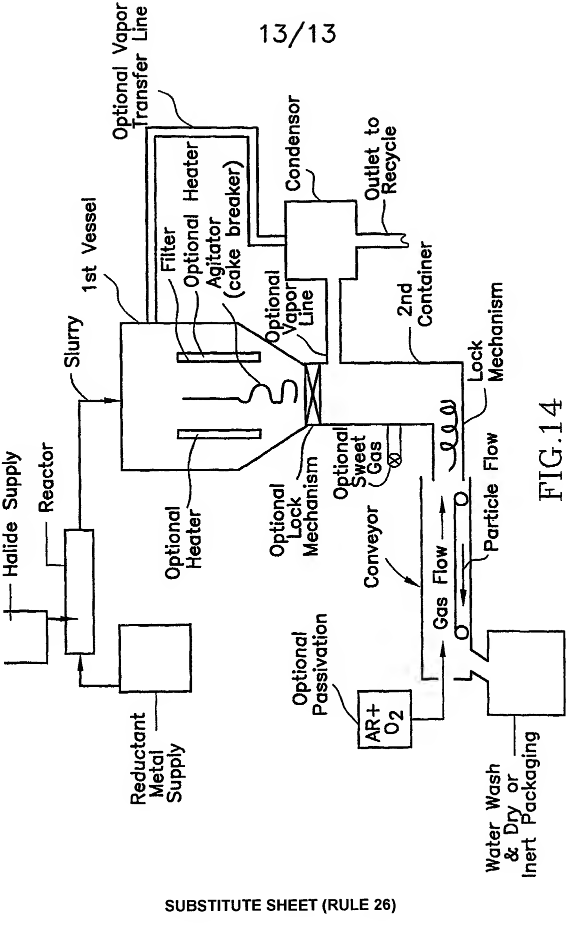

Halide Supply
Reactor
Reductant Metal Supply
Slurry
1st Vessel
Optional Vapor Transfer Line
Filter
Optional Heater
Agitator (cake breaker)
Optional Heater
Optional Vapor Line
Optional Lock Mechanism
Condensor
Outlet to Recycle
2nd Container
Lock Mechanism
Optional Sweet Gas
Conveyor
Gas Flow
Particle Flow
Optional Passivation
AR+ O_2
Water Wash & Dry or Inert Packaging

FIG.14